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THE EFFECTS OF HYDROGEN ISOTOPES AND HELIUM ON THE FLOW AND FRACTURE PROPERTIES OF 21-6-9 STAINLESS STEEL.

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DISCLAIMER

THE EFFECTS OF HYDROGEN ISOTOPES AND HELIUM ON THE TENSILE PROPERTIES OF 21-6-9 STAINLESS STEEL (U)*

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Introduction

High-energy-rate-forged (HERF) stainless steels are used as the materials of construction for tritium and deuterium reservoirs. Hydrogen and helium, the decay product of tritium, are known to embrittle stainless steels (1-4). The resistance to hydrogen and helium induced embrittlement is relatively good for HERF stainless steels when compared to annealed stainless steels due to their high number density of dislocations, which act as traps for hydrogen and helium. However, the degree of the embrittlement in these materials can vary considerably because of microstructure and yield strength variations introduced during the forging process.

In this study the effect of hydrogen and tritium on the room temperature tensile properties of 21-6-9 stainless steel was measured as a function of HERF yield strength in the range of 500 to 918 MPa. The effect of a microstructure was studied also by conducting tensile tests with both HERF samples and annealed samples.

Experimental Procedure

Tensile tests were conducted using samples machined from 21-6-9 stainless steel forgings that were supplied in the form of forward extruded cylinders. The compositions and forging treatments are given in Table I. The forgings were approximately 10 cm long and 3.8 cm in diameter. The tensile samples had a 19.1 mm gage length and a 4.8 mm diameter. The forgings had been HERF'ed to produce nominal yield strengths of 660, 760, 870, and 930 MPa. A few samples from the 930 MPa forging were subsequently annealed at 1144 K for 5 minutes to produce a recrystallized microstructure having a nominal yield strength of 517 MPa. The HERF and annealed microstructures are shown in Figure 1.

One set of samples was exposed to hydrogen at 623 K and 69 MPa for 6 weeks. This treatment saturated the samples with approximately 9500 atomic parts per million (appm) hydrogen based on available diffusivity and solubility data (4). Another set of samples was exposed to tritium

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gas at 423 K and 31 MPa for 9 months, and aged for 12 more months at 298 K for helium build-in from tritium decay. The average tritium and helium concentrations were calculated to be 2470 appm and 390 appm respectively. The samples were pulled at room temperature in air using a screw-driven testing machine and a crosshead speed of 0.0085 mm/s.

Results

The yield strengths, ultimate strengths, percent elongation, and ductility (percent reduction-in-area) of uncharged, hydrogen-charged, and tritium-charged-and-aged samples are summarized in Table II. Yield strengths ranged from 500 MPa for annealed samples to 918 MPa for the highest strength HERF samples. Hydrogen-charged samples had higher yield strengths and ultimate strengths than uncharged samples. The yield strength increase caused by hydrogen was dependent on the initial strength and ranged from about 11% for the 500 MPa samples to 5% for the 918 MPa samples. Tritium-exposed-and-aged samples had even higher strengths. The yield strength increase with respect to uncharged samples ranged from 25% for the 500 MPa samples to 10% for the 918 MPa samples. These observations can be seen in the true stress-true strain curves shown in Figure 2. The tritium-exposed-and-aged samples contain only 25% of the hydrogen isotope concentration as the hydrogen charged samples; however, they also contained 390 appm helium from tritium decay. Thus, on a "per-atom" basis, the results indicate that helium has a larger effect on strength than hydrogen.

The effect of yield strength on ductility of uncharged, hydrogen-charged, and tritium-charged-and-aged is plotted in Figure 3. This figure includes data from annealed and HERF samples. The ductility of uncharged HERF samples was not strongly dependent on yield strength; i.e., ductility was reduced by about 11% as the HERF yield strength was increased from 712 to 918 MPa. Over this same range of HERF yield strengths, the samples containing 9500 appm hydrogen showed on average 40% lower ductilities than the unexposed samples. The HERF tritium-charged-and-aged samples averaged 33% lower ductilities than the unexposed samples over the same range of strengths.

Samples that were annealed prior to tritium charging had much lower ductilities than similarly charged HERF samples (Figure 4). Annealed samples failed prior to necking with ductilities less than 15 percent compared to all HERF samples failed after necking with ductilities greater than 30 percent.

The fracture appearances of HERF and annealed samples are shown in Figures 5 and 6. Unexposed samples and all HERF samples charged with either hydrogen or tritium failed by a strain-controlled microvoid nucleation and growth process. In contrast, annealed samples that were subsequently tritium-charged-and-aged fractured along grain and twin boundaries in a brittle manner.

Discussion

The susceptibility of 21-6-9 austenitic stainless steel to hydrogen and helium effects is dependent on processing and microstructural variables. In this study, the specific effects of microstructure and yield strength on the hydrogen and tritium compatibility of 21-6-9 stainless steel were investigated. The results show that while hydrogen-charged and tritium-charged-and-aged 21-6-9 stainless steel had higher strengths and lower ductilities than uncharged 21-6-9 stainless steel, ductility was not strongly affected by yield strength. The results also show that the resistance to helium embrittlement was strongly affected by microstructure. HERF samples containing tritium and helium failed in a ductile manner; annealed samples containing tritium and helium failed in a brittle manner. These observations indicate that the dislocations in the HERF microstructure help to minimize helium embrittlement by trapping helium away from the grain boundaries. Transmission Electron microscopy examinations are planned to determine if the helium bubble distributions are different in the annealed and HERF microstructures.

The results of this study indicate the importance of microstructural variations on the tritium compatibility of stainless steels. For example, the results suggest that the recrystallized grains near the heat-affected zones of welds in tritium reservoirs may be degraded more by helium than regions away from the welds. This will be a subject of future study.

Conclusions

1. Hydrogen-charged and tritium-charged-and-aged 21-6-9 stainless steel had higher yield strengths and lower ductilities than uncharged 21-6-9 stainless steel. The ductility was not strongly affected by yield strength.

2. A recrystallization annealing heat treatment increased the ductility of uncharged and hydrogen-charged samples, but reduced the ductility of tritium-charged-and-aged samples.

3. HERF 21-6-9 stainless steel was much less susceptible to helium embrittlement than annealed 21-6-9 stainless steel. The dislocation networks in the HERF microstructure apparently act as trap sites for helium and help prevent intergranular fracture.

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TABLE I
Heat Compositions and Forging Treatments

Heat Compositions (w/o)										
Heat[a]	Cr	Ni	Mn	P	Si	C	S	N	O(ppm)	Al
1	19.2	7.22	9.23	.014	.41	.032	.003	.28	19	.002
2	19.4	6.40	8.50	.021	.33	.040	<.001	.28	22	<.001
3	20.1	6.50	9.10	.019	.59	.037	<.001	.29	10	.001

[a] Heat 1 was used for forgings of nominal strengths of 660 and 760 MPa. Heat 2 was used for forgings of nominal yield strengths of 760 and 870 MPa. Heat 3 was used for forgings of nominal yield strengths of 930 MPa.

Forging Conditions: Extrusion die and stub punch; heat parts to 1255 K +/- 10 K; hold for 10 to 15 minutes; forge 1 blow at 2 MPa +/- .17 MPa; water quench; heat to 1116 K +/- 10 K; forge 1 blow at 5.5 MPa +/- .17 MPa; water quench.

Annealing Conditions (Nominal Yield Strength of 517 MPa): 1144 K for 5 minutes.

TABLE II
Summary of Tensile Results
HERF 21-6-9 Stainless Steel

Condition	Nominal Strength (MPa)	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)	Ductility (%)
Uncharged					
Annealed	517	500	811	68.6	74.8
HERF	662	712	932	34.3	71.1
HERF	758	819	969	22.5	56.1
HERF	869	825	1029	28.4	64.3
HERF	931	918	1032	39.5	63.3
Hydrogen Charged					
Annealed	517	555	839	71.1	59.7
HERF	662	776	974	29.1	42.8
HERF	758	1005	1093	23.5	33.0
HERF	869	836	948	----	----
HERF	931	965	1073	38.9	39.2
Tritium Charged and Aged					
Annealed	517	627	782	16.5	13.7
HERF	662	829	996	34.0	46.1
HERF	758	863	1019	21.2	47.7
HERF	869	882	1025	23.8	35.1
HERF	931	1013	1094	37.2	42.8

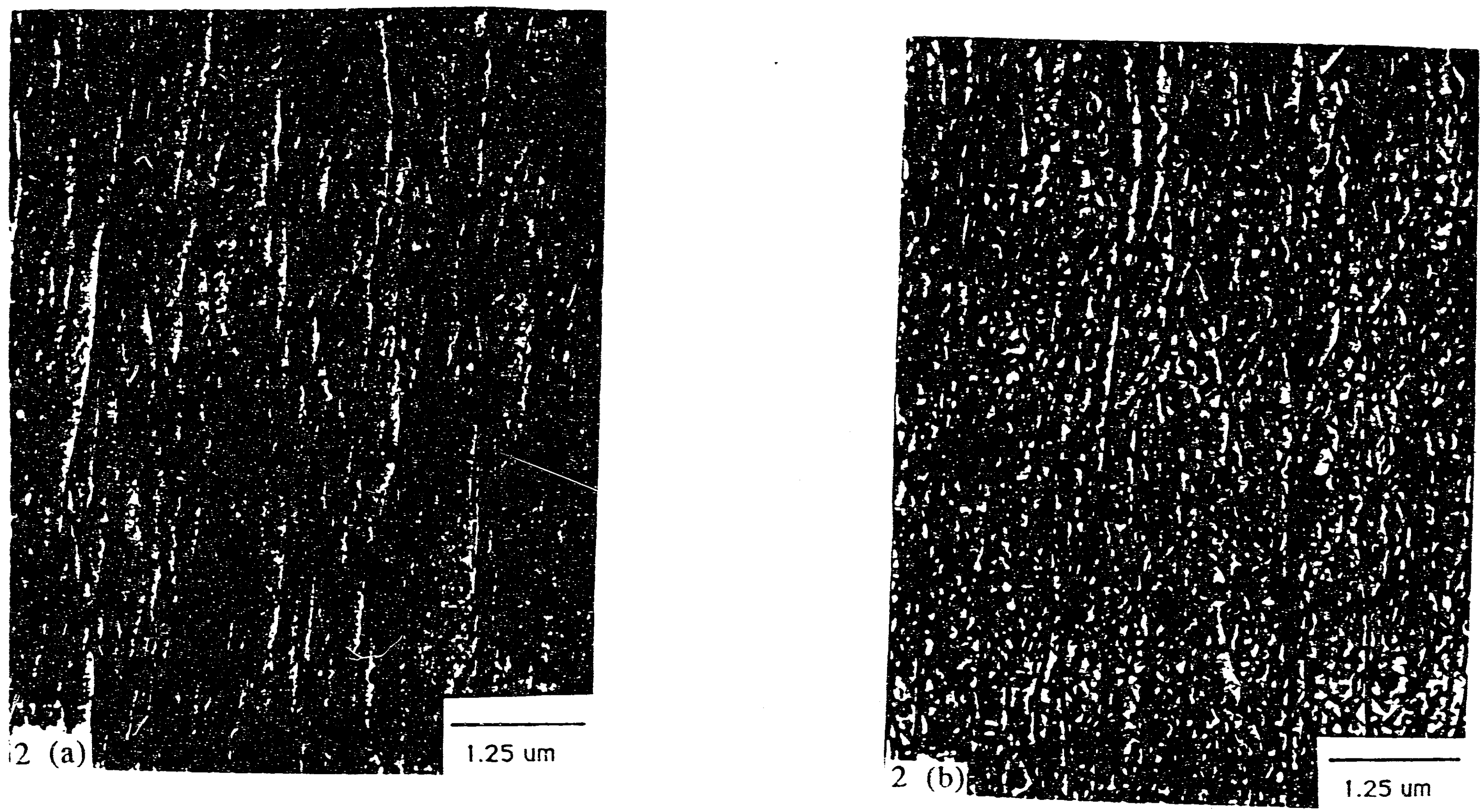


Figure 2. Longitudinal Section Microstructures from Forward Extruded High-Energy-Rate-Forgings (a) HERF; (b) Annealed

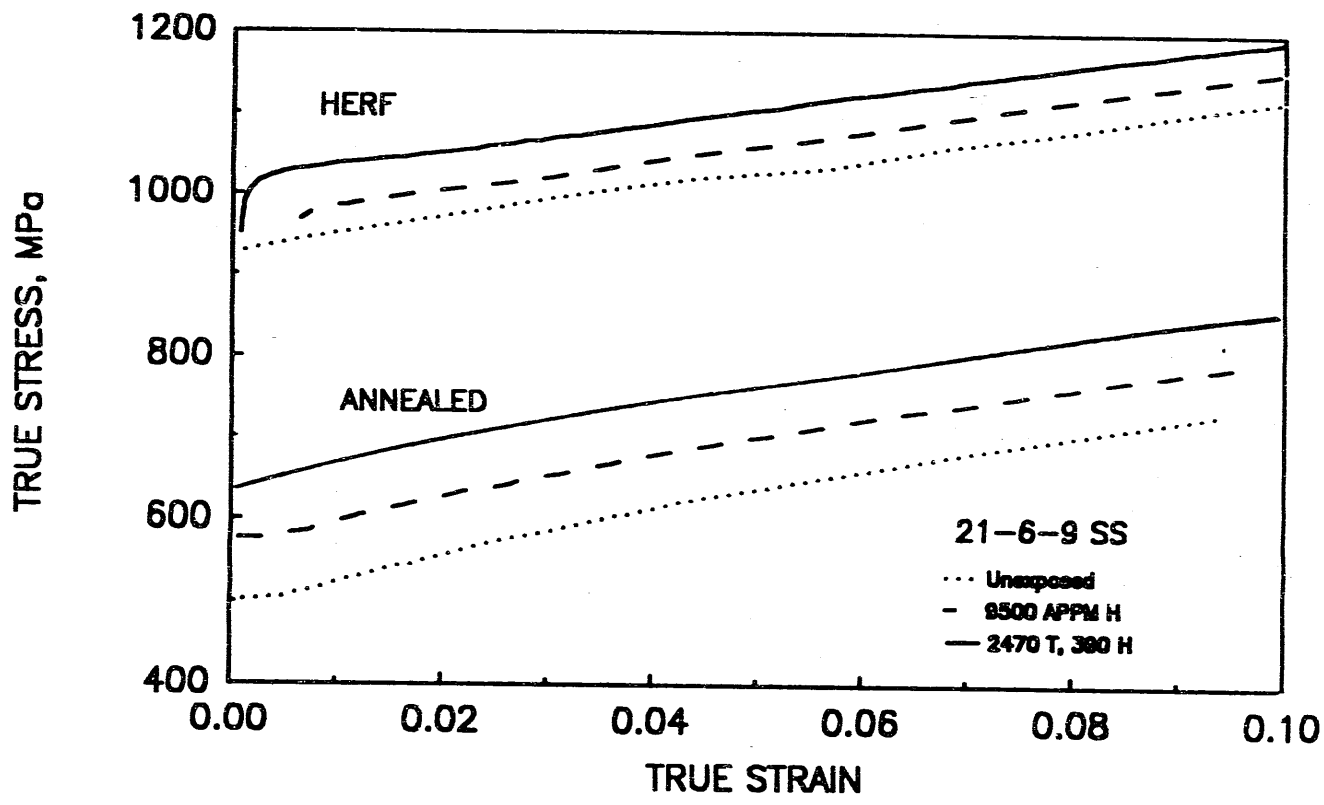


Figure ~~1~~ 2. True Stress-True Strain Curve Showing Significant Strengthening Occurring for Hydrogen-Exposed and Tritium-Exposed-and-Aged Samples. The Strength Increase Caused by Hydrogen and Helium Is Similar in Both Annealed and HERF Samples.

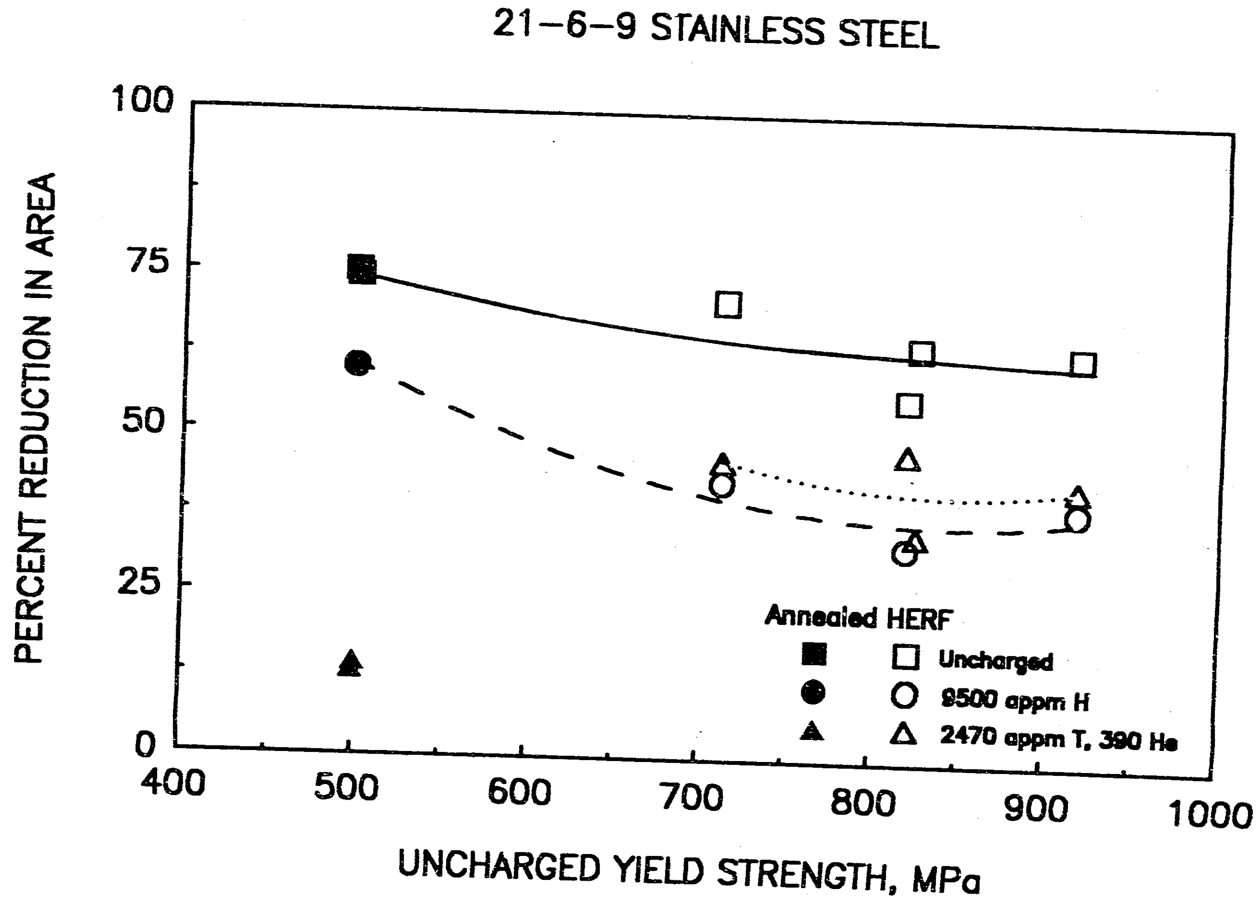
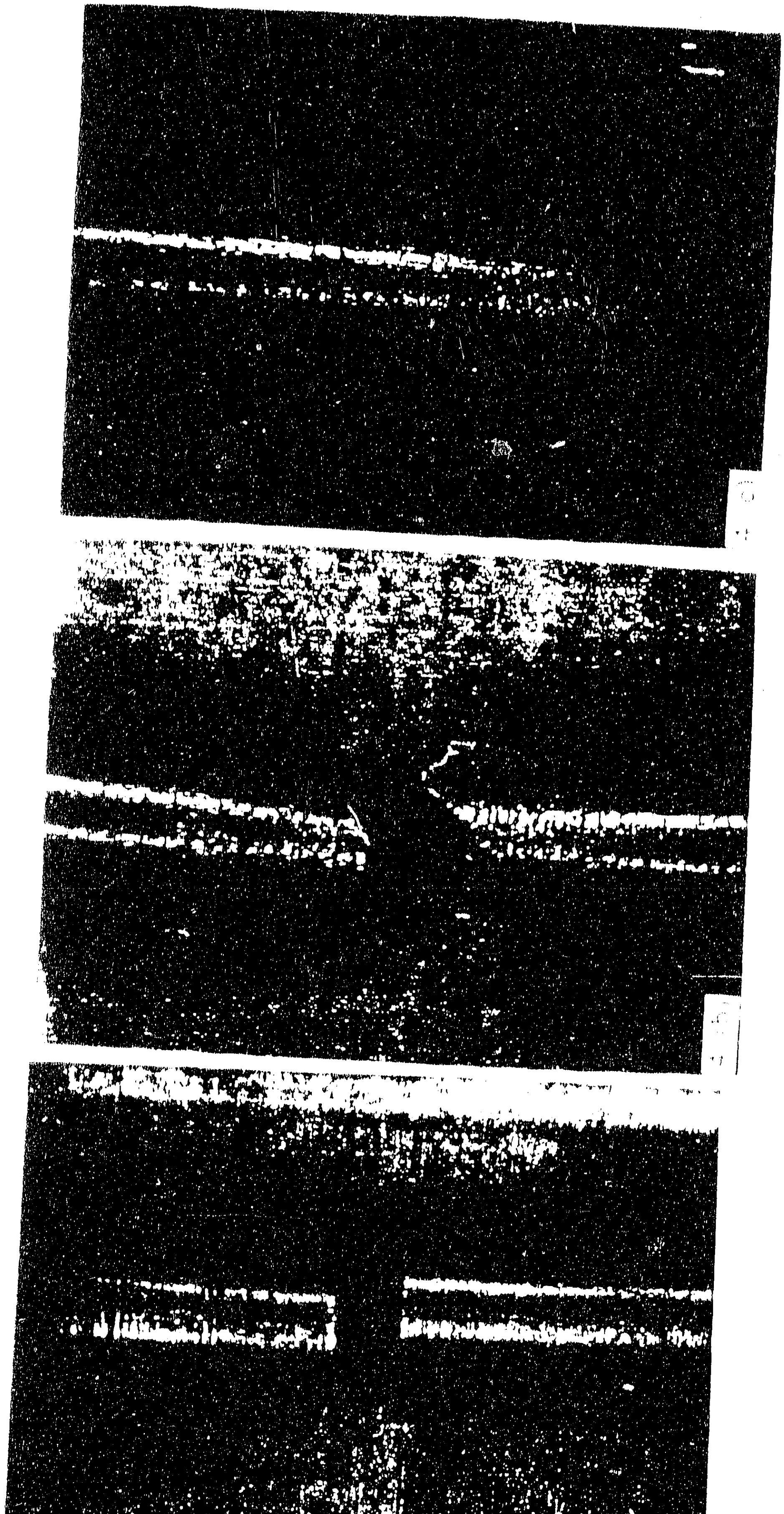


Figure 3. Ductility of Unexposed, Hydrogen-Exposed and Tritium Exposed-and-Aged Samples as a function of the Yield Strength of Unexposed Samples.



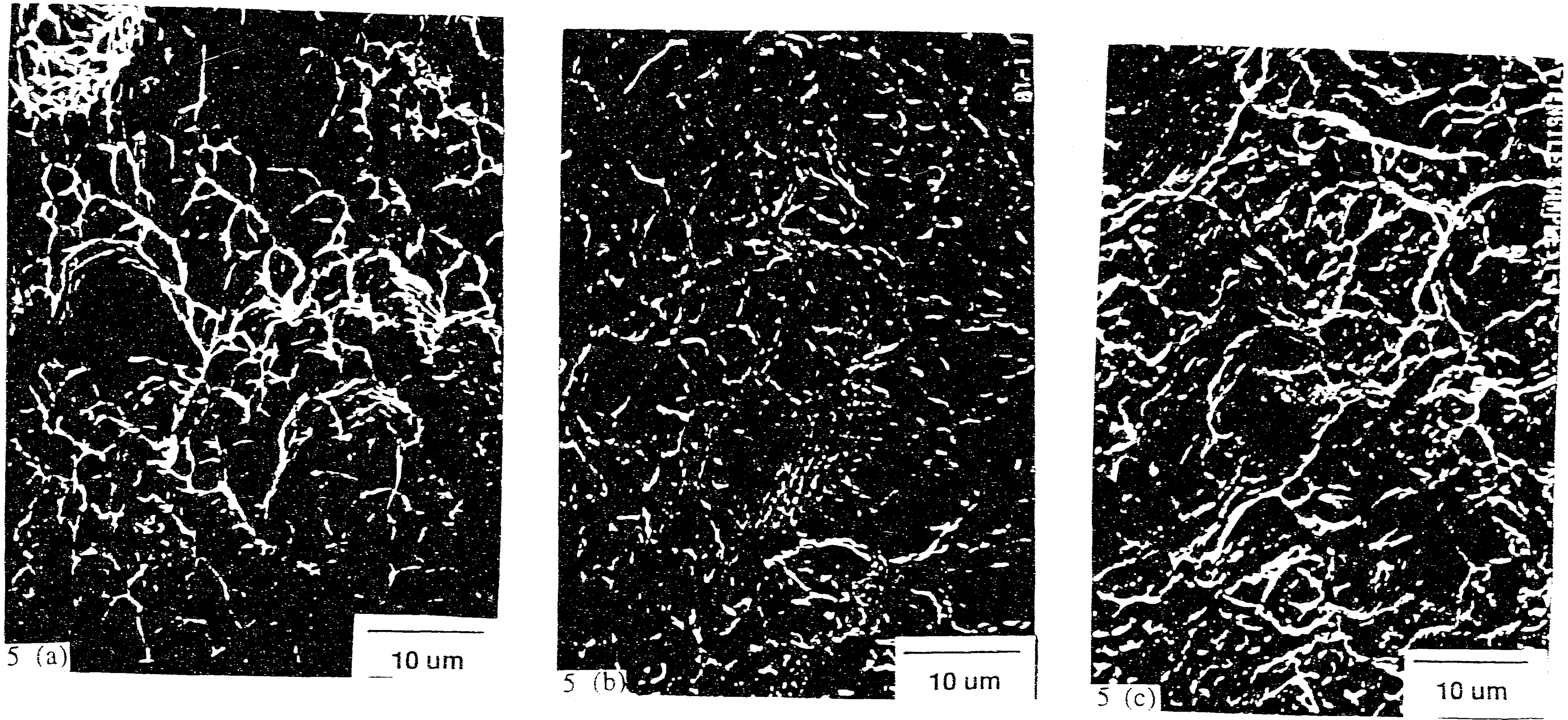


Figure 5. Fracture Appearance of (a) HERF Unexposed; (b) HERF Hydrogen Exposed; and (c) HERF Tritium-Exposed-and-Aged.

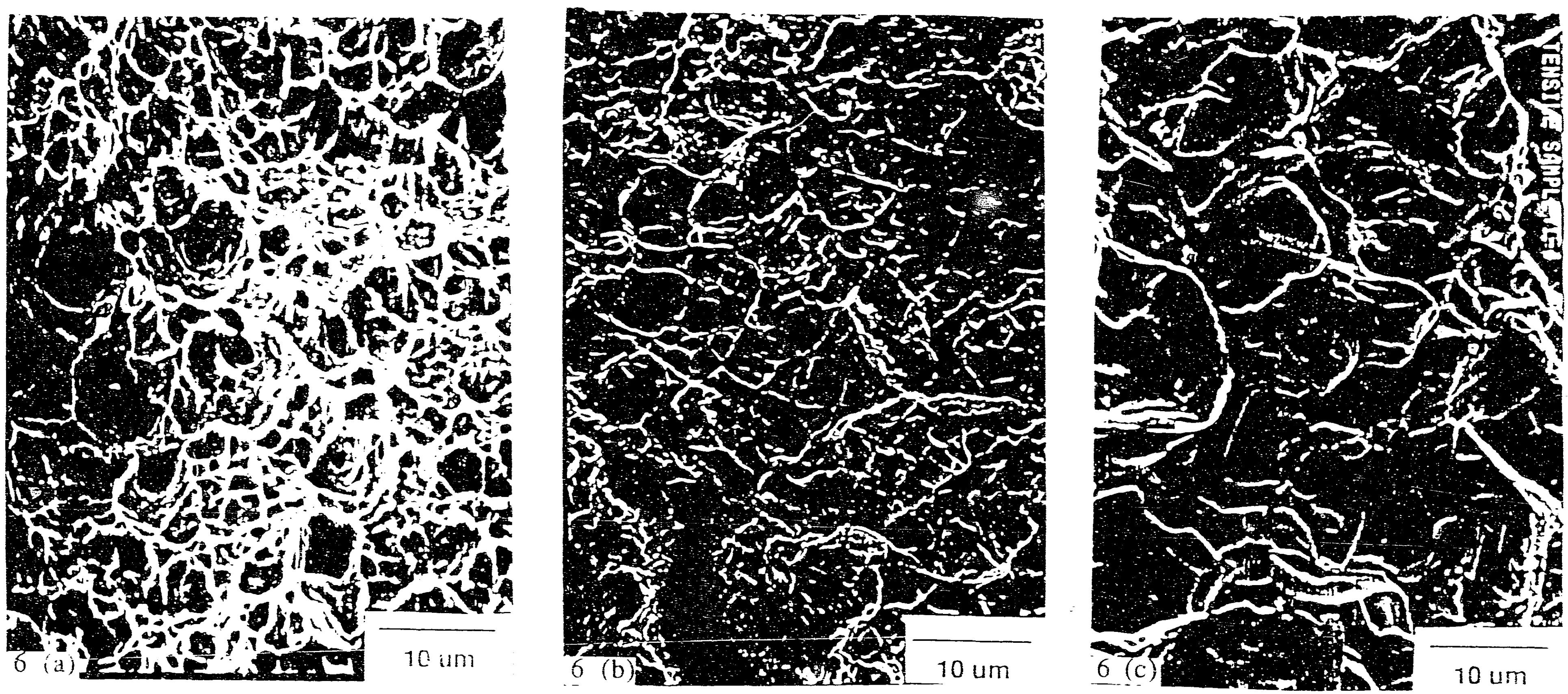


Figure 6. Fracture Appearance of (a) Annealed Unexposed; (b) Annealed Hydrogen Exposed; and (c) Annealed Tritium-Exposed-and-Aged.

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